

Figure 1

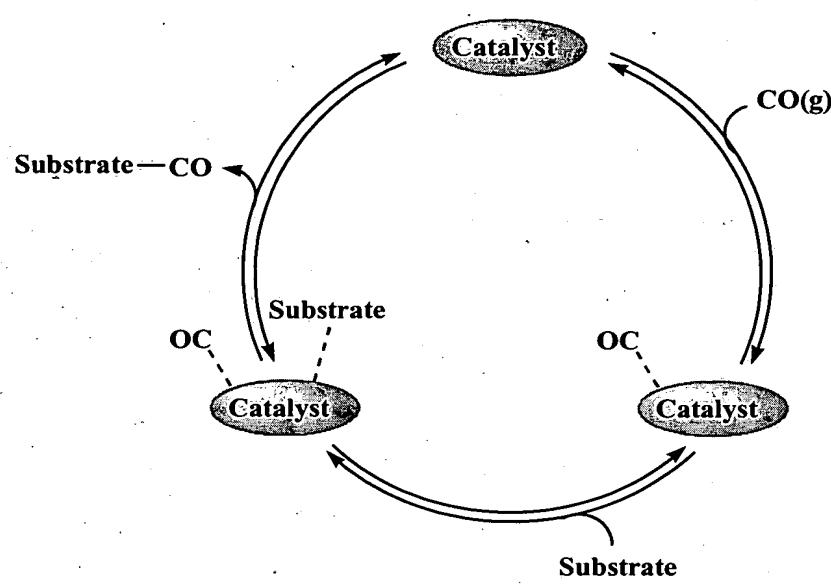
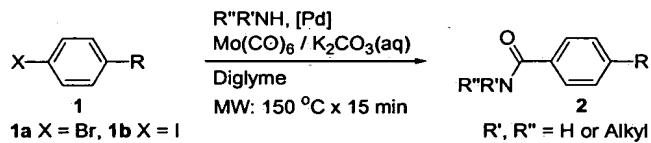
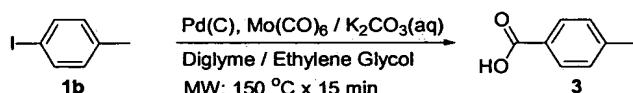


Figure 2



## Microwave-assisted palladium-catalyzed amidation utilizing in situ generated carbon monoxide from $\text{Mo}(\text{CO})_6$ .



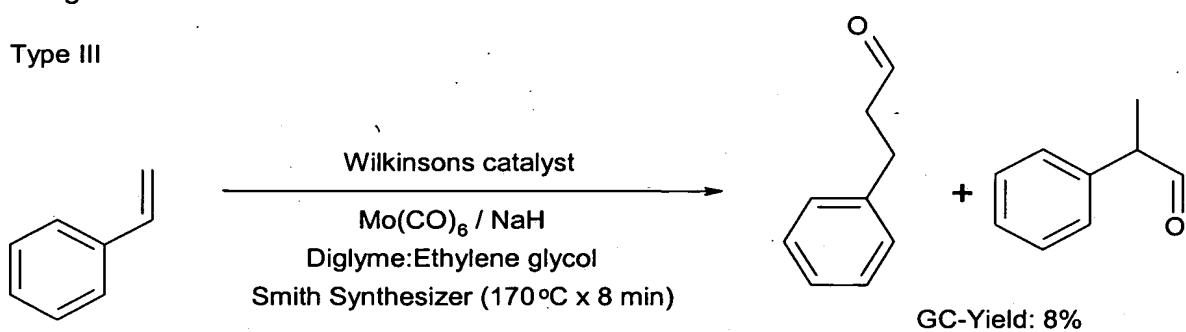
## Microwave-assisted palladium-catalyzed generation of *p*-methyl benzoic acid from tolyl iodide utilizing *in situ* generated carbon monoxide from $[\text{Mo}(\text{CO})_6]$ .

<sup>a</sup>Average isolated yields from 2-3 runs (0.23 mmol scale, SmithSynthesizer™, >95% by GC/MS). <sup>b</sup>p-Methyl-benzoic acid. Ethylene glycol was added as co-solvent.

entry, aryl-X	R-group	nucleophile	product	yield <sup>a</sup> (%)
1, <b>1a</b>	MeO-	<i>n</i> -BuNH <sub>2</sub>	<b>2a</b>	70
2, <b>1a</b>	Me-	<i>n</i> -BuNH <sub>2</sub>	<b>2b</b>	71
3, <b>1a</b>	F <sub>3</sub> C-	<i>n</i> -BuNH <sub>2</sub>	<b>2c</b>	75
4, <b>1a</b>	Ac-	<i>n</i> -BuNH <sub>2</sub>	<b>2d</b>	77
5, <b>1a</b>	MeO-	Piperidine	<b>2e</b>	65
6, <b>1a</b>	Me-	Piperidine	<b>2f</b>	66
7, <b>1a</b>	F <sub>3</sub> C-	Piperidine	<b>2g</b>	74
8, <b>1a</b>	Ac-	Piperidine	<b>2h</b>	83
9, <b>1a</b>	Me-	Benzyl amine	<b>2i</b>	48
10, <b>1b</b>	MeO-	<i>n</i> -BuNH <sub>2</sub>	<b>2a</b>	69
11, <b>1b</b>	Me-	<i>n</i> -BuNH <sub>2</sub>	<b>2b</b>	72
12, <b>1b</b>	F <sub>3</sub> C-	<i>n</i> -BuNH <sub>2</sub>	<b>2c</b>	78
13, <b>1b</b>	Ac-	<i>n</i> -BuNH <sub>2</sub>	<b>2d</b>	79
14, <b>1b</b>	MeO-	Piperidine	<b>2e</b>	66
15, <b>1b</b>	Me-	Piperidine	<b>2f</b>	69
16, <b>1b</b>	F <sub>3</sub> C-	Piperidine	<b>2g</b>	75
17, <b>1b</b>	Ac-	Piperidine	<b>2h</b>	76
18, <b>1b</b>	Me-	Water	<b>3</b>	87 <sup>b</sup>

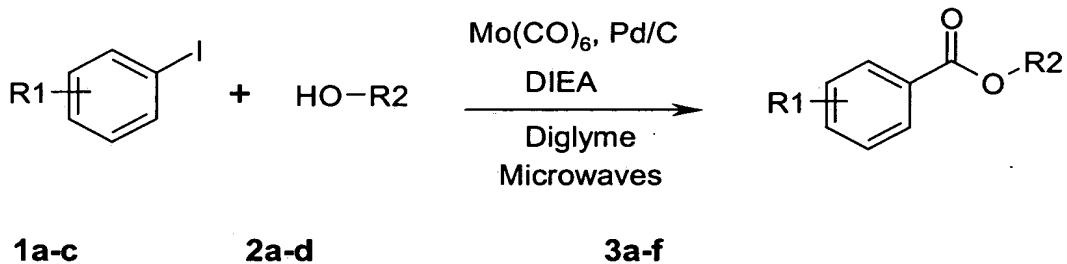
Figure 3

Type III



100-0752-17403

Figure 4



<b>R1</b>	<b>R2</b>	<b>Time (s)</b>	<b>Conversion of 1a</b>	<b>Isolated Yields (%) of 3</b>
<b>1a</b> 4-OMe	<b>2a</b> -nBu	300	90%	<b>3a</b> 75%
<b>1b</b> 2-Me	<b>2a</b> -nBu	300	Full	<b>3b</b> 46%
<b>1a</b> 4-OMe	<b>2b</b> -tBu	900	Full	<b>3c</b> 38%
<b>1a</b> 4-OMe	<b>2c</b> -CH <sub>2</sub> Ph	900	b	<b>3d</b> 36%
<b>1a</b> 4-OMe	<b>2d</b> -CH <sub>2</sub> CH <sub>2</sub> Si(Me) <sub>3</sub>	900	Full	<b>3e</b> 65%
<b>1c</b> 4-CF <sub>3</sub>	<b>2d</b> -CH <sub>2</sub> CH <sub>2</sub> Si(Me) <sub>3</sub>	900	Full	<b>3f</b> 65%

<sup>a</sup>Measured with GC-MS on crude products. <sup>b</sup>not detected with GC-MS.

Figure 5

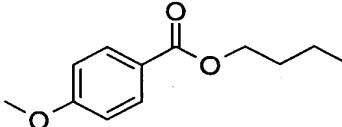
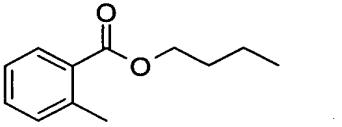
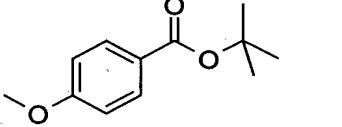
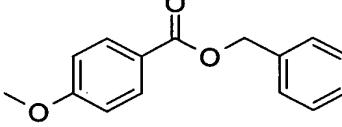
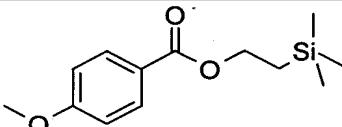
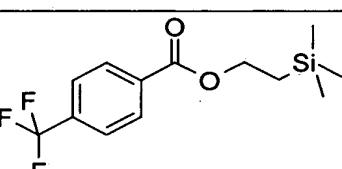
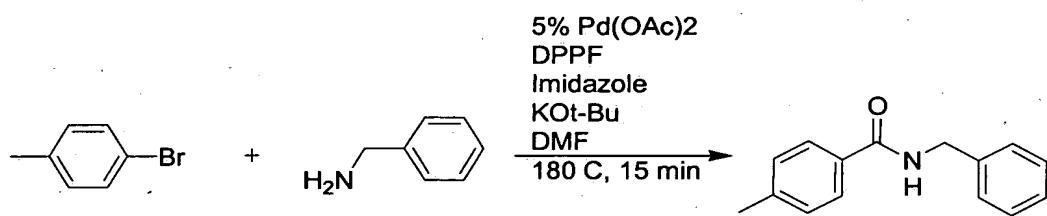
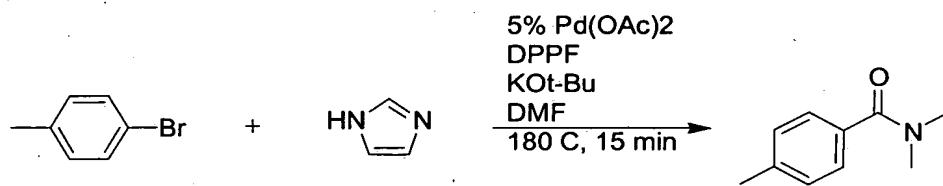
Number	Structure	Name
3a		Butyl-4-methoxybenzoate
3b		Butyl-4-methylbenzoate
3c		t-Butyl-4-methoxybenzoate
3d		Bensyl-4-methoxybenzoate
3e		(2-trimethylsilyl)ethyl-4-methoxybenzoate
3f		(2-trimethylsilyl)ethyl-4-trifluoromethylbenzoate

Figure 6



**4-Acetyl-*N*-*n*-butyl-benzamide (2d).** White crystals.  $^1\text{H}$  NMR (19 °C, TMS): δ 7.90 (d, 2H; Aryl), 7.77 (d, 2H; Aryl), 6.4 (bs, 1H; CONH), 3.39 (q, 2H; N-CH<sub>2</sub>), 2.45 (s, 3H; COCH<sub>3</sub>), 1.54 ppm (m, 2H; CH<sub>2</sub>), 1.33 (m, 2H; CH<sub>2</sub>), 0.89 (t, 3H; CH<sub>3</sub>);  $^{13}\text{C}$  NMR (CDCl<sub>3</sub>, 25 °C, TMS): δ 197 (CO), 166 (CONH), 138.9 (C-ipso), 138.7 (C-ipso), 128 (C-HAryl), 127 (CHAryl), 40 (C-aliphatic), 31 (C-aliphatic), 27 (C-aliphatic), 20 (C-aliphatic), 14 (C-aliphatic). MS (70 eV): m/z (%): 219 (10) [M<sup>+</sup>], 177 (25), 147 (100). Elemental Analysis: Calculated for C<sub>13</sub>H<sub>17</sub>NO<sub>2</sub>: C, 71.2; N, 6.4; H, 7.8; Found: C, 71.6; N, 6.3; H, 7.9.

**4-Trifluoromethylphenyl-piperidin-1-yl-methanone (2g).** Yellow oil.  $^1\text{H}$  NMR (19 °C, TMS): δ 7.66 (d, 2H; Aryl), 7.48 (d, 2H; Aryl), 3.75 (bs, 2H; CH<sub>2</sub>), 3.32 (bs, 2H; CH<sub>2</sub>), 1.67 (bs, 4H; CH<sub>2</sub>), 1.52 (bs, 2H; CH<sub>2</sub>);  $^{13}\text{C}$  NMR (25 °C, TMS): δ 168 (CO), 140 (C-ipso), 131 (q; CF<sub>3</sub>), 127 (CHAryl), 126 (CHAryl), 122 (C-ipso), 49 (broad, C-aliphatic), 43 (broad, C-aliphatic), 27 (broad, C-aliphatic), 26 (broad, C-aliphatic), 24 (C-aliphatic). MS (70 eV): m/z (%): 256 (80) [M+-1], 173 (100), 145 (75). Elemental Analysis: Calculated for C<sub>13</sub>H<sub>14</sub>F<sub>3</sub>NO × ½H<sub>2</sub>O: C, 58.6; N, 5.3; H, 5.7; Found: C, 58.8; N, 5.1;